PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.: 10/263,135 Confirmation No.: 2687
Applicant(s): Fritzemeier et al.
Filed: October 2, 2002
Art Unit: 1742
Examiner: Jenkins, Daniel J.
Title: METHOD FOR PREPARING CRYOMILLED ALUMINUM ALLOYS AND COMPONENTS EXTRUDED AND FORGED THEREFROM

Docket No.: 038190/234776
Customer No.: 00826

October 27, 2004

Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.131

Sir:

We, Daniel E. Matejczyk and Thomas J. Van Daam, hereby declare and state that:

1. We are inventors of the claimed invention of the above-identified U.S. Patent Application Serial No. 10/263,135.

2. At least as early as December 31, 2001, we reduced to practice our invention as described and claimed in the subject application, generally directed to a method of preparing cryomilled aluminum alloys and components extruded and forged therefrom.

3. Exhibit A (attached) is a copy of the "Nanophase Processing Yield/Scrap Tally" spreadsheet maintained by Raul Perez, working under the supervision of Dan Matejczyk. The spreadsheet shows that alloys were cryomilled as batch numbers NPCxx-05 and NPCxx-06 (portions of batch numbers have been redacted) at least as early as December 31, 2001. Cryomilling is a nanostructured material synthesis technique. Exhibit B (attached) shows handwritten lab notes, dated at least as early as December 31, 2001.

which indicate the composition of the inert-gas-atomized starting powders that were weighed out and then cryomilled in an attritor. The composition of the metal powder, as indicated, was approximately 8 wt% Mg with the balance of the composition being approximately 92 wt% Al. 8 wt% Mg is well within the 1 atomic% to 11 atomic% range recited in the pending claims of the 10/263,135 application. As indicated on Exhibit A, both samples NPCxx-05 and NPCxx-06 were degassed in Impeller can #3, also designated as "Imp 3". Exhibit C (attached) is a copy of lab notebook pages that evidence the degassing and consolidation procedure applied to the contents of Impeller can #3. Exhibit D (attached) is a copy of hand-written notes showing the extrusion of the resulting consolidated billet from Impeller can #3 at least as early as December 31, 2001. Exhibit E (attached) is a printout of laboratory results, dated at least as early as December 31, 2001, showing the Mg and Fe content of the extruded sample (Imp 3). Finally, Exhibit F (attached) is a copy of test results, dated at least as early as December 31, 2001, showing that the nitrogen content of the extruded sample (Imp 3) is above 0.3 wt%. The resultant nitrogen content indicated at least a 0.3 wt% increase in nitrogen content compared to the alloy powder prior to cryomilling. Dates, personal information, and other information not relevant to the substantiation of invention have been redacted from the copies included in the Exhibits. Although the dates have been redacted, each of Exhibits C, D, E, and F is dated at least as early as December 31, 2001.

4. During production of the Imp 3 sample described above, refractory material was not added to the alloy by anyone associated with production of the alloy. It would have been customary and regular practice to record any additions of refractory materials to the alloy in the cryomilling logs. The lack of any indication, in the cryomilling logs, that refractory material was added is positive evidence that no refractory material was added to the Imp 3 sample.

5. The attached exhibits evidence that, at least as early as December 31, 2001, we reduced to practice a cryomilled aluminum alloy by the steps of: providing a metal

powder comprising 89 atomic% to 99 atomic% aluminum, 1 atomic% to 11 atomic% of a secondary metal selected from the group consisting of magnesium, lithium, silicon, titanium, zirconium, and combinations thereof; and processing the metal powder with a nanostructured material synthesis technique such that at least 0.3 weight% nitrogen was added to the metal; wherein refractory material was not added to the metal during processing. The exhibits further illustrate that we cryomilled such a powder, removed gaseous components from the cryomilled powder, consolidated the cryomilled powder into a metallic billet, and extruded the metallic billet, at least as early as December 31, 2001.

6. We hereby declare that all statements made herein of our own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issued thereon.

Daniel E. Matejczyk 10/27/04
Daniel E. Matejczyk

Thomas J. Van Daam 10/27/04
Thomas J. Van Daam

CLT01/4675634v3

Nanophase Processing Yield/Scrap Talley

Run No.	Date	Yield (Kg)	Yield %	Scrap (Kg)	Pwdr in Attrit	Attrit Run'g Tot	Comments
[illegible]	[illegible]	[illegible]	51%	2.995	[illegible]	[illegible]	[illegible]

Example 5

REDACTED

NPC	05	19.625	98%	2.650	-2.275	12.590	R.H 2%.
NPC	06	15.660	78%	3.010	+1.330	13.920	R.H. 4%. Packed impeller can #3 with 6/2 & 6/4 run.

REDACTED

oleanic acid.

NPC -05

Powder Measure

5000g
5000g
5000g
2000g
17000g

} C.P. Al Batch #9-8035

in hopper

8:25 AM hopper on
8:32 LN_2 Thru hopper
9:00 Powder drop
9:10 @ 100 rpm
5:10 PM Powder dum
52° in glove box

3000g } 50/50 Al-Mg Batch #9-7024S

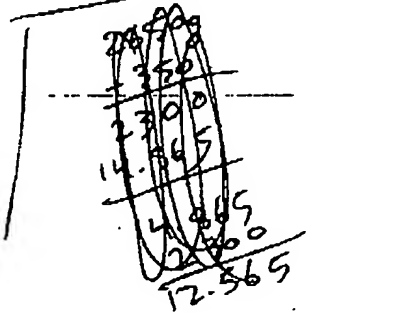
40g Stearic Acid

R.H Φ → 2%

yield	scrap
3705g	2650g
4265g	
4205g	
4845g	
2605g	
19625	

19625
2650
22275

20



BEST AVAILABLE COPY

NPC -06

Powder Measure

5005 g
5105 g
4890 g
2000 g
17000g

} C.P. Al Batch #9-8035

3000 g 50/50 Mg-Al Batch # 9-7024S

40g Stearic Acid

7^{35} AM	in hopper
7^{45}	hopper on
7^{51}	LN_2 Thru hopp
8^{07}	Powder drop
8^{14}	@ 100 rpm
4^{14}	Powder dump
4^{23}	in glove box

yield
4250 g
4310 g
4380 g
2720 g
15660 g

scrap
3010 g

R.H. 4%

42 **TITLE** Degas of Impeller can #3 PROJEC. NO. Nanophase Al
BOOK NO.

Work continued from Page

The boulder was brought into the large glovebox and Can and welded shut on . The can was packed on 33.785 kg.

8:20 AM Can was connected to vacuum pump.

8:30

Time	Vac	I	Pwr	rpm	Tpump	T_{c_1}	T_{c_2}	Furnace temp/set	High limit
8:33	4×10^{-2} (crossing)	2.4	116W	38k	36°C				
8:45	2.05×10^{-4}	1.7	69W	38°K	39°C				
8:53	9.81×10^{-5}	1.5	51W	38K	40°C				
9:00	6.85×10^{-5}	1.3	43W	38K	40°C	(Chiller switched on)			
9:40	1.51×10^{-5}	1.1	32W	38k	34°C				
8:50	2.88×10^{-5}	1.0	30W	38K	33°C				

9:20 · Vacuum level ~ 3.00×10^{-5} ⇒ There's a leak.
· Did a helium leak check, couldn't find a leak.
· Moving the 'O' ring results in slight vacuum changes. ⇒ Possible leak
· Did a 5 min nitrogen purge before exchanging ring for a new one.

9:35 · open to high vacuum after changing "O" ring, and position of "O" Ring

Work continued to Page 43

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

SIGNATURE Daniel Perez DATE

DISCLOSED TO AND UNDERSTOOD BY DATE WITNESS DATE

TITLE Degas of Impeller Can #3 **PROJECT NO.** Nanophase Al **43**
BOOK NO.

Work continued from Page 42

Time	Vac	I	Pwr	rpm	Tpump
9:00	3.73×10^{-2} (Roughing)	2.4	111	38K	43°C
10:15	1.23×10^{-2} (Roughing)	2.0	84	38K	43°C
10:30	5.08×10^{-4}	1.7	64	38K	42°C
10:45	2.69×10^{-4}	1.4	50	38K	40°C
11:00	1.89×10^{-4}	1.3	45	38K	39°C
11:15	1.41×10^{-4}	1.3	41	38K	38°C
11:25	Helium check for leaks				
11:30	1.05×10^{-4}	1.2	38	38K	37°C
11:35	9.69×10^{-5}	1.2	37	38K	37°C
11:45	8.16×10^{-5}	1.2	36	38K	37°C
11:50	7.78×10^{-5}	1.1	35	38K	37°C

- completed helium check @ 11:40, no leaks detected
- Vacuum is pumping too slow. N_2 back purge again (5 min) and moved "O" again. Plug replaced "O" ring.

	open to high Vacuum once again				
	1.62×10^{-2} (Roughing)	2.1	92	38K	46°C
	7.82×10^{-4}	2.9	81	38K	45°C

a/ Roughing Pump @ 1.00×10^{-2}

Work continued to Page 44

DATE

Read and Understood by DATE WITNESS DATE

44 **TITLE** Degas of impeller can #3 **PROJECT NO.** Nanophase A1

BOOK NO.

Work continued from Page 43

Time	Vac	I	Pwr	rpm	Temp
13:00	4.78×10^{-4}	1.7	63W	38K	43°C
13:15	2.86×10^{-4}	1.5	53W	38K	42°C
13:30	1.78×10^{-4}	1.4	46W	38K	40°C
13:45	1.30×10^{-4}	1.3	41W	38K	40°C
14:00	9.48×10^{-5}	1.2	37W	38K	39°C
14:15	7.76×10^{-5}	1.1	35W	38K	38°C
14:30	6.33×10^{-5}	1.1	33W	38K	38°C
14:45	5.41×10^{-5}	1.1	32W	38K	37°C
15:00	4.91×10^{-5}	1.0	31W	38K	36°C
15:15	4.55×10^{-5}	1.0	31W	38K	36°C
15:30	4.35×10^{-5}	1.0	30W	38K	36°C
15:45	4.17×10^{-5}	1.0	30W	38K	35°C
16:00	3.97×10^{-5}	1.0	29W	38K	35°C
16:15	3.87×10^{-5}	1.0	29W	38K	35°C
16:30	3.75×10^{-5}	1.0	29W	38K	35°C

N_2 Back Purge (5 min)

Work continued to Page 45

SIGNATURE Lail Pierce DATE

TITLE Degas of impeller can #3 **PROJECT NO.** Nanophase A1 **BOOK NO.** **45**

Work continued from Page 44

Time	Vac	I	Pwr	rpm	Temp
16:45	6.89×10^{-1} (Roughing)	5.4	346 W	38 K	42°C
17:00	2.00×10^{-2} (Roughing)	2.2	98 W	38 K	45°C
17:15	4.55×10^{-4}	1.7	62 W	38 K	43°C

• Helium check entire system for leaks

7:20	3.85×10^{-5}	1.0	30 W	38 K	33°C

• N_2 back purge
• Changed and moved 'O' ring again.

8:05 evacuation tube connected to high vacuum.

8:30	2.90×10^{-3} (Roughing)	2.3	107 W	38 K	44°C
8:45	6.78×10^{-3} (Roughing)	1.9	75 W	38 K	43°C
9:00	3.59×10^{-3}	1.5	52 W	38 K	40°C
9:45	1.13×10^{-4}	1.2	39 W	38 K	38°C
10:15	7.16×10^{-5}	1.2	34 W	38 k	37°C
12:30	3.74×10^{-5}	1.1	32 W	38 K	42°C

Disconnected chiller

Work continued to Page 46

SIGNATURE Daniel Pinkas DATE

READ AND UNDERSTOOD BY DATE WITNESS DATE

46 **TITLE** Degas of impeller can #3 **PROJECT NO.** Nanophase A1

BOOK NO.

Work continued from Page 46

Time	Vac	I	Air	rpm	Tamb
15:35	3.73×10^{-5}	1.1	32W	38K	44°C

~~15:20 Helium probe to can to check for leaks~~

~~Drop in vacuum~~

Try to get helium detector to work. Not possible.

14:30	3.93×10^{-5}	1.0	30W	38K	45°C

No improvement in vacuum reading. => Definite leak

MassSpec Leak detector used to find a small leak in side weld. The side weld was rewelded in 2 spots

12:26	Evacuation began				
13:46	2.55×10^{-4}	1.8	70W	38K	43°C
14:00	6.79×10^{-5}	1.2	40W	38K	38°C
14:45	6.23×10^{-5}	1.0	29W	38K	34°C
18:35	1.00×10^{-4}	1.0	28W	38K	34°C

Good vacuum reading => no more leaks.

9:40	6.09×10^{-7}	1.1	30W	38K	33°C
10:40	6.05×10^{-7}	1.1	30W	38K	33°C

N_2 Gas Purge (5 min)

Work continued to Page 47

SIGNATURE Paul Kelly DATE

READ AND UNDERSTOOD BY DATE WITNESS DATE

TITLE Degas of impeller can #3 **PROJECT NO.** Nanophase Al **BOOK NO.** 47

Work continued from Page 46

Time	Vac	I	Pwr	rpm	Temp	T_{c1}	T_{c2}	Furnace temp/set	High Limit
11:30	1.70×10^{-4}	1.4A	49W	38K	40°C				
13:00	7.09×10^{-6}	1.1A	33W	38K	35°C				
14:00	1.71×10^{-6}	1.0A	29W	38K	35°C				
14:30	1.15×10^{-6}	1.0	29W	38K	35°C				
	N_2 Gas back purge (5 min)								
15:45	1.52×10^{-4}	1.4	49W	38K	42°C				
17:05	1.10×10^{-5}	1.0	29W	38K	37°C				
	HOT EVACUATION STARTED / 3 DAY RAMP TO 600°F								
8:50	3.97×10^{-7}	1.0	26W	38K	35°C	80	80	150/20	
~~8:55~~								~~150/20~~ 150 hold/15min	
9:10	4.33×10^{-7}	1.0	27W	38K	35°C	118.2	118.8		163
9:25	5.45×10^{-7}	1.0	27W	38K	35°C	120.4	118.6	158/25 (30m)	147
9:55	7.59×10^{-7}	1.0	27	38	35	138.4	135.8	216/ramp 15m hold 215	206
10:10	9.41×10^{-7}	1.0	27	38	36	146.6	143.0	214/ramp 300 30m	205
10:40	1.70×10^{-6}	1.0	27	38	36°C	185.0	179.2	300/hold 300 60m	290
11:40	7.48×10^{-6}	1.0	27	38	37°C	300	291	300/ramp 350	291
12:10								350 hold 60m	

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page 48

SIGNATURE Dan Z Dimmick DATE

48 TITLE Degas of impeller can #3 PROJECT NO. Nanophase A1
BOOK NO.

Work continued from Page 47

Time	Vac	I	Pwr	rpm	Temp	Tc1	Tc2	Temp/Set	H2O Unit
12:30	1.77×10^{-5}	1.0A	27W	38K	37°C	268.6	258.6	350	341
13:10	2.59×10^{-5}	1.0A	28W	38K	38°C	277.8	273.0	ramp to 375 30min	342
13:40	2.98×10^{-5}	1.0A	28W	38K	38°C	293.6	289.4	375 hold 30m	367
14:25								ramp to 400 30min	376
14:32	3.24×10^{-5}	1.0A	28W	38K	38°C	309.8	306.2	ramp	376
14:55	3.15×10^{-5}	1.0A	28W	38K	38°C	321.8	318.4	400 hold	394
16:00	2.86×10^{-5}	1.0A	28W	38K	37°C	337.8	334.8	400 hold	395
17:00	2.46×10^{-5}	1.0A	28W	38K	36°C	346.2	343.8	400 hold	395
8:20	8.69×10^{-6}	1.0	28W	38K	33°C	384.2	382.8	400 Hold	399
8:30	8.71×10^{-6}	1.0	28W	38K	33°C	384.2	383.0	450/30	399
9:45	8.90×10^{-6}	1.0	29W	38K	32°C	385.8	384.6	450/30	445
9:00	8.91×10^{-6}	1.0	29W	38K	32°C	390.6	389.0	450 hold 30	454
9:15	9.46×10^{-6}	1.0	29W	38K	32°C	394.4	392.6	450/hold	448
9:30	1.0 1.10^{-5}	1.0	29W	38K	32°C	397.0	395.0	500/30	448
9:45	1.10×10^{-5}	1.0	29W	38K	32°C	405.2	403.2	500/30	475

Work continued to Page 49

SCIENTIFIC SINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

SIGNATURE Daniel Perry DATE

DISCLOSED TO AND UNDERSTOOD BY DATE WITNESS DATE

TITLE impeller can #3

PROJECT NO.

BOOK NO.

49

Work continued from Page 48

time	Voc	I	Pwr	rpm	Temp	Tc1	Tc2	Furnace temp/set	High limit
10:00	1.22×10^{-5}	1.0	29	38K	32°C	415.2	413.2	500/hold	497
10:15	1.42×10^{-5}	1.0	29 W	38 K	31°C	421.4	419.4	500/hold	495
10:30	1.69×10^{-5}	1.0	29 W	38 K	31°C	426.2	424.2	525/30	495
10:45	1.98×10^{-5}	1.0	29 W	38 K	31°C	432.4	430.2	525/30	508
11:00	2.42×10^{-5}	1.0	29 W	38 K	31°C	441.8	439.6	525/hold	521
11:15	2.78×10^{-5}	1.1	29 W	38 K	31°C	449.4	448.6	525/hold	519
11:30	3.19×10^{-5}	1.1	29 W	38 K	31°C	449.4	447.4	525/hold	520
11:45	3.63×10^{-5}	1.1	29 W	38 K	31°C	453.2	451.4	525/hold	525
12:00	4.21×10^{-5}	1.1	30 W	38 K	31°C	463.8	462.0	525/hold	565
12:15	4.69×10^{-5}	1.1	30 W	38 K	31°C	467.0	465.4	525/hold	565
12:30	5.69×10^{-5}	1.1	30 W	38 K	31°C	470.6	469.0	550/30	563
12:45	6.28×10^{-5}	1.1	31 W	38 K	31°C	476.6	475.2	550/30	580
13:00	7.23×10^{-5}	1.1	31 W	38 K	30°C	484.4	483.4	550/overtemp	596
13:15	8.33×10^{-5}	1.1	31 W	38 K	30°C	490.0	488.6	550/hold	565
13:30	9.88×10^{-5}	1.1	32 W	38 K	30°C	493.6	492.2	550/hold	564
13:45	1.19×10^{-4}	1.2	33 W	38 K	31°C	496.8	495.4	550/hold	563

Work continued to Page 50

SIGNATURE [signature] DATE

DISCLOSED TO AND UNDERSTOOD BY DATE WITNESS DATE

50 TITLE impeller can #3

PROJECT NO.

BOOK NO.

Work continued from Page 49

Time	Vac	I	Pwr	RPM	Tpump	Tc1	Tc2	Furnace temp/set	High limit
14:00	1.44×10^{-4}	1.2	33 W	38 K	31° C	498.2	497.8	550/hold	563
14:15	1.74×10^{-4}	1.2	34 W	38 K	31° C	506.2	500.0	550/hold	563
14:30	2.07×10^{-4}	1.3	35 W	38 K	31° C	503.0	501.8	550/hold	563
	Roughing Pump	1.97×10^{-3}							
14:45	2.48×10^{-4}	1.3	36 W	38 K	31° C	505.0	503.4	550/hold	562
	Roughing Pump	4.43×10^{-3}							
15:00	2.91×10^{-4}	1.3	39 W	38 K	32° C	506.2	504.8	550/hold	562
	Roughing Pump	7.58×10^{-3}							
15:15	3.36×10^{-4}	1.4	41 W	38 K	32° C	507.4	506.2	550/hold	562
	Roughing Pump	1.31×10^{-2}							
15:30	5.03×10^{-4}	1.5	45 W	38 K	32° C	508.8	507.6	550/hold	562
	Roughing Pump	2.32×10^{-2}							
15:45	2.70×10^{-4}	1.7	54 W	38 K	33° C	510.2	508.8	550/hold	561
	Roughing Pump	4.72×10^{-2}							
16:00	3.04×10^{-4}	1.8	62 W	38 K	33° C	510.8	509.6	550/hold	561
	Roughing Pump	7.49×10^{-2}							
16:15	4.46×10^{-4}	2.0	73 W	38 K	34° C	511.8	510.2	550/hold	561
	Roughing Pump	1.23×10^{-1}							
16:30	6.29×10^{-4}	2.2	85 W	38 K	35° C	512.6	511.2	550/hold	561
	Roughing Pump	1.84×10^{-1}							
16:45	8.80×10^{-4}	2.4	99 W	38 K	36° C	513.4	511.8	550/hold	561
	Roughing Pump	2.47×10^{-1}							
17:00		2.5	108 W	38 K	37° C	514.0	512.6		560
		Roughing Pump	3.05×10^{-1}			; Ion gauge @ 16:52			
17:15		2.6	115 W	38 K	38° C	514.8	513.2	550/hold	560
		Roughing Pump	3.54×10^{-1}						
17:30		2.7	121 W	38 K	39° C	515.4	513.8		561
		Roughing Pump	3.95×10^{-1}						

Work continued to Page 51

DATE

Raul Perez

READ AND UNDERSTOOD BY DATE WITNESS DATE

TITLE impeller can #3

PROJECT NO. **51**

BOOK NO.

Work continued from Page 50

Time	Vac	I	Pwr	rpm	Tpump	Tc1	Tc2	Furnace temp/set	High limit
17:45		2.7	122 W	38 K	39° C	516.0	514.4	550/hold	560
		Roughing Pump 3.96×10^{-1} → PEAK							
17:00		2.7	120 W	38 K	40° C	516.6	515.0	550/hold	560
		Roughing Pump 3.84×10^{-1}							
18:15		2.6	119 W	38 K	40° C	517.0	515.4	550/hold	560
		Roughing Pump 3.57×10^{-1}							
18:30		2.5	112 W	38 K	40° C	517.4	515.8	550/hold	560
		Roughing Pump 3.12×10^{-1}							
18:45		2.3	105 W	38 K	40° C	517.8	516.2	550/hold	560
		Roughing Pump 2.65×10^{-1}							
8:45	7.59×10^{-5}	1.1	33 W	38 K	33° C	522.4	520.8	575/30	559
9:00	7.52×10^{-5}	1.1	32 W	38 K	33° C	525.6	524.4	575/30	575
9:15	7.70×10^{-5}	1.1	32 W	38 K	33° C	530.8	529.4	575/hold	589
9:45	8.27×10^{-5}	1.1	32 W	38 K	34° C	535.6	534.4	575/hold	587
10:19	8.83×10^{-5}	1.1	33 W	38 K	34° C	538.4	537.2	575/hold	587

Work continued to Page 52

SIGNATURE John Z. Zimmerman DATE

WITNESS DATE

52 **TITLE** impeller can #3 **PROJECT NO.**

BOOK NO.

Work continued from Page 51

	V&C	I	Pwr	rpm	T_{pump}	T_{C_1}	T_{C_2}	Furnace Temp/Set	High Limit
10:45	8.99 $\times 10^{-5}$	1.1	33W	38k	35°C	539.6	541	610/30	587
11:15	9.90 $\times 10^{-5}$	1.1	33W	38k	35°C	554.4	553.4	610/hold	630
13:00	1.52 $\times 10^{-4}$	1.2	37	38k	37°C	569.6	568.4	610/hold	623
	roughing pump	6.86 $\times 10^{-3}$							
14:15	1.69 $\times 10^{-4}$	1.3	37	38k	38°C	573.6	572.4	625/30	621
	roughing pump	8.14 $\times 10^{-3}$							
15:10	1.70 $\times 10^{-4}$	1.3	38	38k	38°C	583.4	582.2	625/Local	638
	roughing pump	9.22 $\times 10^{-3}$							
16:00	1.72 $\times 10^{-4}$	1.3	38	38k	38°C	587.2	586.0	625/Local	637
17:00	1.59 $\times 10^{-4}$	1.3	37	38k	38°C	589.8	588.8	625/Local	636
	roughing pump	7.50 $\times 10^{-3}$							
09:00	2.49 $\times 10^{-5}$	1.0	28W	38k	35°C	594.0	592.4	625/Local	635
09:10	2.52 $\times 10^{-5}$	1.0	28W	38k	35°C	594.0	592.6	632/Local	635
09:30	2.55 $\times 10^{-5}$	1.0	28W	38k	35°C	596.8	595.2	632/Local	643
10:30	2.57 $\times 10^{-5}$	1.0	28	38k	34°C	598.6	597.2	632/Local	643
12:00	2.47 $\times 10^{-5}$	1.0	28	38	34°C	599.6	598.2	632/Local	642
15:00	2.20 $\times 10^{-5}$	1.0	27	38	37°C	600.6	599.2	632/Local	642
17:38	1.93 $\times 10^{-5}$	1.0	28W	38k	36°C	600.8	599.6	632/Local	642

Work continued to Page 53

SIGNATURE [signature] DATE

READ AND UNDERSTOOD BY DATE WITNESS DATE

53

TITLE Impeller can #3

PROJECT NO.

BOOK NO.

Work continued from Page 52

Time	Vac	I	Pwr	rem	Temp	Tc	Tcb	Furnace temp/set	high limit
9:30	9.60×10^{-6}	1.0	27 W	38 K	34°C	600.4	549.0	632/Local	642
:30	2.85×10^{-6}	1.0	28 W	38 K	32°C	600.0	598.6	632/Loc	641
:41	2.55×10^{-6}	1.0	26 W	38 K	32°C	600.2	598.8	632/Loc	641
	• Shut furnace off and chiller								
:00	1.33×10^{-6}	1.0	27 W	38 K	33°C	479.6	475.6		
:10	7.73×10^{-7}	1.0	26 W	38 K	34°C	444.4	445.8		
:20	2.81×10^{-7}	0.9	26 W	38 K	34°C	400.6	402.2		
:25	1.45×10^{-7}	0.9	26 W	38 K	35°C	372.6	374.6		
:30	6.05×10^{-8}	0.9	25 W	38 K	35°C	311.6	312.2		
:00	5.36×10^{-8}	0.9	25 W	38 K	35°C	275.2	250.6		
	• Thermo couples disconnected								
	• Tube crimped.						(Ultimate Vacuum 9.56×10^{-9} @ 400 hours)		
:05	5.12×10^{-8}	0.9	25 W	38 K	35°C				
	• Tube welded								
	• Vacuum level jumped all around and reached @ 7.01×10^{-8}								
:08	5.12×10^{-8}								

Work continued to Page 54

DATE

DATE WITNESS DATE

54 TITLE Impeller can #3 PROJECT NO. Nanophase Al

BOOK NO.

Work continued from Page 53

Final Mass of can 33.665 Kg

Tube cut off 0.045 Kg

33.710

Initial can Mass 33.7665 Kg

* Tube was cut 3 times.

Can was shipped out on

9-28-

Work continued to Page

SCIENTIFIC GINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

SIGNATURE D and Z. Summit DATE

READ AND UNDERSTOOD BY DATE WITNESS DATE

IMP1
4 INDUCERS (9, 10, 11)(12)
IMP2
SHIPPED IMP3 & IMP4 ON AUG 20 (MID SEPT. EXTRUSION)
* FINAL FURNACE SETPOINT WAS 371°F
IMP2 LOADED INTO FURNACE @ 8:00am.

TIME BILLET TEMP T/C #2 FURNACE TEMP #3

3/12 PREHEAT BILLET LOADED @ 10:30 pm
1:30 pm + EXTRUSION

°F

°F

R E D A C TE D

FURNACE LOAD 3:45am @ 400°F
200°F @ 4:10 am
350°F @ 6:00am
380°F @ 7:00am

BREAK THROUGH F TONS = 4237 TONS

IMP 3 & 4

BILLET TEMPS

	T/C #2 FURNACE	#4	T/C #3	
7:00am.		7 (BILLET 3)	6	FURNACE SETPOINT DROPPED FROM 410 → 400
7:15am	389.2	388.2	400.5	BILLET 4 9-30-98 - IMP 4 [TORN JACKET]
	389.1	390.2	400.3	
		392.8	400.8	... FROM 400 → 376°F
7:30am.	389.3		399.4	
7:40am	389.3	391.6	386.2	... FROM 376 → 369°F
8:45am	380.8	386.2		... 8:50 369 → 364
9:25am.	377.1	382.3	381.0	
9:30am	372.2	378.5	376.5	364 → 368
	371	378.0	375.	LINER 634°F
← 9:46	370.	375.	377 (DIE 200 → HEAT TO 400°F) @ 9:30	
		374		

PULLED FIRST.
950 FIRST EXTRSN

10:30

BEST AVAILABLE COPY

THE BOEING COMPANY
Rocketdyne Propulsion
And Power System
MATERIALS APPLICATION
PROCESS
LABORATORY OPERATIONS

DATE
G.O. 61918
REFERENCE EWR#936081
REPORT No. 9811-4
PROGRAM NAME Nanophase Aluminum
QUANTITY 2
MATERIAL Aluminum Alloy
SPECIFICATION
SUBMITTED BY R. Perez
DEPT. 912
LOCATION CANOGA
PHONE 3958

TEST REPORT

LABORATORY TEST RESULTS

SAMPLE		Mg %/wt	Iron %/wt
9/30	IMP3	8.8	0.1
9/30	IMP4	8.6	0.1

Log No. 9811-4
Charge No. 24410-61918-98100
Notebook Page No. 2054-24

W. Ho
ANALYST

DATE

A. Apeti (W. Ho)
APPROVED

DATE

LECO CORPORATION
3000 Lakeview Avenue
St. Joseph, Michigan 49085-2396

Work Order No: 12536
Classification: S
PO/WO No NR98439623
Customer No 2593
Account No: 109485

CUSTOMER SERVICE ANALYSIS REPORT

Customer: BOEING NORTH AMERICAN, INC.
ROCKETDYNE DIVISION/633 CANOGA AVE.
CANOGA PARK CA 91303

Attention: SONYA REID Dept. 917, MC BA71

The following is our laboratory analysis report on samples submitted by your company.

Any additional details of this analysis report can be obtained from the Technical Services Laboratory. Call 616-982-2277.

NOTE: We request that all samples submitted to the LECO® Technical Services Laboratory be accompanied by a Material Safety Data Sheet (MSDS) describing each material's characteristics. This information permits proper handling and storage.

Results for Sample: 63681 IMP3 *Al7.5%Mg*

Instrument: CS444

Standard: NIST 125b @ 0.028% C
Remarks: Preheated Crucible 528-018, Preheated Lid 528-043, ~1 g Lecocel II HP 502-173, ~ 1 g Iron Chip 502-231
Preparation: Abraded with a file, sectioned with a shear, rinsed in acetone and dried with warm air.

% Sulfur	% Carbon
	0.152
	0.151
	0.148

Instrument: RH402

Standard: Hydrogen Gas Dose
Remarks: 769-761 Crucible
Preparation: Abraded with a file, sectioned with a shear, rinsed in acetone and dried with warm air.

Hydrogen ppm	Bulk ppm	Surface ppm
43.09	42.78	0.311

Instrument: TC436

Standard: LECO Std. 501-644 @ 0.0107% O, 0.0071% N (J0215-2)
Remarks: 782-720 Crucible, 1 g Nickel Basket 502-344, 0.05 g Graphite Powder 501-073
Preparation: Abraded with a file, sectioned with a shear, rinsed in acetone and dried with warm air.

% Nitrogen	% Oxygen
0.383	0.239
0.381	0.235